



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 10

1200 Sixth Avenue  
Seattle, Washington 98101


IN REPLY

REFER TO: OEA-095

January 11, 1999

MEMORANDUM

SUBJECT: Bunker Hill, CLP Metals Analysis, Data Validation  
Case: 26610  
SDG: MJAE38

FROM:   
Laura Castrilli, Chemist  
Quality Assurance and Data Unit, OEA



TO: Mary Kay Voytilla, Regional Project Manager  
Office of Environmental Cleanup

CC: Bruce Woods, Region 10 CLP TPO  
Jim Stefanoff, CH2M Hill

The following is a validation of ICP-AES and mercury analyses of two water samples from the Bunker Hill project. The analyses were performed following the USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis Multi-media, Multi-Concentration, ILM04.0. Analyses were conducted by Sentinel Inc. of Huntsville, Alabama. This validation was conducted for samples MJAE38 (total/unfiltered) and MJAE39 (dissolved/filtered).

**Data Qualifications**

The following comments refer to the Sentinel Laboratory's performance in meeting quality control specifications outlined in the *CLP Statement of Work (CLP-SOW) for Inorganic Analysis, rev. ILM04.0*. The comments presented herein are based on the information provided for the review.

**1.0 Timeliness - Acceptable**

The technical (40 CFR part 136) holding time from the date of collection for mercury in water is 28 days. The holding time for the remaining metals in water is 180 days. The samples were collected on 11/20/98. Mercury analyses were completed on 12/07/98. ICP-AES analyses were completed on 12/09/98.

**2.0 Sample Preparation - Acceptable**

The samples were prepared for mercury analysis on 12/04/98. The samples were prepared for ICP-AES analysis on 12/02/98.

January 11, 1999

must be between 80% and 120%. All ICS recoveries associated with reported sample results were within the recovery criterion. The samples' raw data was checked. None of the samples had interfering levels of analytes and therefore no qualification was made on this basis.

#### **7.0 Duplicate Analysis - Acceptable**

Duplicate analyses were done on both the dissolved and filtered samples. Water duplicate results were within the  $\pm 20\%$  Relative Percent Difference (RPD) or  $\pm$ CRDL criteria for water results  $< 5$  times the CRDL criteria. No qualification was made on this basis.

#### **8.0 Field Duplicate Analysis - Not Applicable**

Field duplicate analysis for samples in this SDG was not indicated in the field collection documentation.

#### **9.0 Matrix Spike Analysis -**

Matrix spike sample analyses are done to provide information about the effect of the sample matrix on digestion and measurement methods. Matrix spike recovery must be within the limits of 75 - 125%.

Matrix spike analyses were done on both the dissolved and total samples. All matrix spike recoveries were within the required QC limits; with the exception of antimony (73.5% for the total sample). The antimony result for the total sample MJAE38 was qualified 'J', estimated (suspected low bias). The laboratory 'N' qualifier was removed from antimony result for the dissolved sample as the spike recovery on the dissolved sample was inside the acceptance criteria.

#### **10.0 Graphite Furnace Atomic Absorption Spec (GFAAS) QC - Not Applicable -**

GFAAS was not used for the analysis of these samples.

#### **11.0 ICP-AES Serial Dilution -**

Both the total and dissolved samples were analyzed by ICP-AES serial dilution to check for potential interferences. All analytes which exceeded the minimum concentration criterion (50 times the IDL) agreed within the 10%D criteria; therefore no qualification was made based on serial dilution.

#### **12.0 Detection Limits - Acceptable**

Sample results which fall below the instrument detection limit (IDL) are assigned the value of the instrument detection limit and the 'U' qualifier is attached.

January 11, 1999

### **3.0 Calibrations/Calibration Verifications - Acceptable**

The samples were analyzed for mercury by CVAAS on 12/07/98. Initial calibration included one blank and five standards. The curve was linear with a correlation coefficient greater than 0.995.

The samples were analyzed by ICP-AES on 12/09/98. The instrument was standardized according to the analytical method using one blank and a single calibration standard for each element.

All ICP-AES and CVAAS (mercury) calibrations were performed as required and met the acceptance criteria; therefore, no qualification was made on this basis.

Calibration verification samples are required before and after sample analysis and after every 10 samples during analysis. Mercury recoveries must be within 80-120%. Other metal recoveries must be within 90-110%.

All ICP-AES and CVAAS (mercury) calibration verification (initial and continuing) samples bracketing reported sample results met the recovery criteria. Calibration verification samples were analyzed after every ten samples. No qualification was made based on ICP-AES or CVAAS calibration verification.

### **4.0 Laboratory Control Samples - Acceptable**

Laboratory Control samples are digested and analyzed along with the samples to verify the efficiency of laboratory procedures. All recoveries associated with reported sample results met the acceptance criteria for control samples.

### **5.0 Blanks - Acceptable**

Procedural blanks were prepared with the samples to show potential contamination from the digestion or analytical procedure. If an analyte was found in the associated blank, the sample results were qualified if the analyte concentration was less than five times the analytical value in the blank.

Thallium was detected in ICP-AES continuing calibration blanks. Potassium in continuing calibration blanks for ICP-AES had negative results with absolute values greater than the detection limit. Associated sample results were greater than five times the associated blank level and were not qualified based on blank contamination.

### **6.0 ICP-AES Interference Check Sample - Acceptable**

The interference check sample (ICS) is analyzed by ICP-AES to verify interelement and background correction factors. Analysis is required at the beginning and end of each sample analysis run and recoveries

January 11, 1999

Contract Required Detection Limit (CRDL) standards are required to demonstrate a linear calibration curve near the CRDL. CRDL standards were run at the required frequency.

### 13.0 Overall Assessment of the Data

This validation of the data is based on the criteria outlined in the *National Functional Guidelines for Inorganic Data Review (02/94)*. One antimony result was qualified based on matrix spike recovery. The data as qualified is acceptable for all purposes.

Below are the definitions for the National Functional Guidelines for Inorganic Data Review (02/94) qualifiers used when validating/qualifying data from Inorganic analysis.

#### DATA QUALIFIERS

- U - The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. (Note: Analyte may or may not be present.)
- UJ - The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

EPA SAMPLE NO.

## INORGANIC ANALYSIS DATA SHEET

MJAE38

Lab Name: SENTINEL, INC.

Contract: 68-D6-0001

Lab Code: SENTIN

Case No.: 26610

SAS No.:

SDG No.: MJAE38

Matrix (soil/water): WATER

Lab Sample ID: 16852S

Level (low/med): LOW

Date Received: 11/21/98

% Solids: 0.0

Concentration Units (ug/L or mg/Kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	457			P
7440-36-0	Antimony	4.1	B	# J	P
7440-38-2	Arsenic	100			P
7440-39-3	Barium	14.5	B		P
7440-41-7	Beryllium	0.60	U		P
7440-43-9	Cadmium	75.1			P
7440-70-2	Calcium	8320			P
7440-47-3	Chromium	0.90	U		P
7440-48-4	Cobalt	13.2	B		P
7440-50-8	Copper	112			P
7439-89-6	Iron	35300			P
7439-92-1	Lead	647			P
7439-95-4	Magnesium	4750	B		P
7439-96-5	Manganese	5660			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	12.5	B		P
7440-09-7	Potassium	1320	B		P
7782-49-2	Selenium	1.9	U		P
7440-22-4	Silver	2.6	B		P
7440-23-5	Sodium	681	B		P
7440-28-0	Thallium	3.6	U		P
7440-62-2	Vanadium	2.9	U		P
7440-66-6	Zinc	19300			P
	Cyanide				NR

Color Before: COLORLESS

Clarity Before: CLEAR

Texture:

Color After: COLORLESS

Clarity After: CLEAR

Artifacts:

Comments:

EPA SAMPLE NO.

## INORGANIC ANALYSIS DATA SHEET

MJAE39

Lab Name: SENTINEL, INC.

Contract: 68-D6-0001

Lab Code: SENTIN

Case No.: 26610

SAS No.:

SDG No.: MJAE38

Matrix (soil/water): WATER

Lab Sample ID: 16853S

Level (low/med): LOW

Date Received: 11/21/98

% Solids: 0.0

Concentration Units (ug/L or mg/Kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	370			P
7440-36-0	Antimony	4.0	U	<del>N</del>	P
7440-38-2	Arsenic	72.1			P
7440-39-3	Barium	14.9	B		P
7440-41-7	Beryllium	0.60	U		P
7440-43-9	Cadmium	68.0			P
7440-70-2	Calcium	8800			P
7440-47-3	Chromium	0.90	U		P
7440-48-4	Cobalt	13.8	B		P
7440-50-8	Copper	96.2			P
7439-89-6	Iron	31000			P
7439-92-1	Lead	620			P
7439-95-4	Magnesium	4850	B		P
7439-96-5	Manganese	5720			P
7439-97-6	Mercury	0.10	U		CV
7440-02-0	Nickel	10.6	B		P
7440-09-7	Potassium	1340	B		P
7782-49-2	Selenium	1.9	U		P
7440-22-4	Silver	2.2	B		P
7440-23-5	Sodium	783	B		P
7440-28-0	Thallium	3.6	U		P
7440-62-2	Vanadium	2.9	U		P
7440-66-6	Zinc	18300			P
	Cyanide				NR

11/25/99

Color Before: COLORLESS

Clarity Before: CLEAR

Texture:

Color After: COLORLESS

Clarity After: CLEAR

Artifacts:

Comments: